# Polymer-Carbon Black Composite Sensors in an Electronic Nose for Air-Quality Monitoring

M.A. Ryan, A.V. Shevade, H. Zhou, and M.L. Homer

### Abstract

An electronic nose that uses an array of 32 polymer—carbon black composite sensors has been developed, trained, and tested. By selecting a variety of chemical functionalities in the polymers used to make sensors, it is possible to construct an array capable of identifying and quantifying a broad range of target compounds, such as alcohols and aromatics, and distinguishing isomers and enantiomers (mirror-image isomers). A model of the interaction between target molecules and the polymer—carbon black composite sensors is under development to aid in selecting the array members and to enable identification of compounds with responses not stored in the analysis library.

**Keywords**: chemical sensors, electronic noses, polymer–carbon black composite sensors

# Introduction

The Jet Propulsion Laboratory's electronic nose (JPL ENose) is an autonomous, miniature device developed for use as an airquality monitor in human habitats in space.1 This device is under development at JPL, where it has been taken from initial work at the California Institute of Technology<sup>2</sup> in developing polymer-carbon black composite sensors to a fully operational device capable of identifying and quantifying 20 organic and inorganic compounds that may be present in recycled air in spacecraft.<sup>1,3</sup> The JPL ENose detects the target compounds at concentrations related to their 24 h spacecraft maximum allowable concentrations (SMACs), exposure levels set by NASA.

The JPL ENose first-generation device was successfully demonstrated in a flight experi-

ment on space shuttle flight STS-95 in 1998,<sup>1,4</sup> since 1998, we have focused on sensor and sensing array optimization,<sup>5</sup> developing a molecular model of sensor response,<sup>7</sup> and on further development of the hardware.

In this article, we focus on the polymer–carbon black composite films that compose the sensing surfaces, the approaches used to select the polymers in the array, and work in developing a molecular model of sensor response. We will also discuss data analysis considerations and how environmental variations are taken into account.

### **Electronic Noses in Space**

Electronic noses have been proposed for many applications within the space program, including as chemical-sensing systems for atmospheric studies on planetary surfaces and for monitoring air quality in human habitats.3 Because electronic noses are low-power devices and can be configured to respond to a broad range of compounds, they are appealing as devices to be used in planetary studies. For planetary exploration, an electronic nose can be used to study the constituents of a planetary atmosphere and atmospheric variations with location or time. To search for evidence of life on other planets, an electronic nose may be used to sample for metabolic products. To achieve the necessary selectivity for such analyses, the sensing media in the array can be chosen to distinguish compounds of similar structure, including isomers and enantiomers (mirror-image isomers).6 Application of electronic noses to atmospheric studies will require the development of methods to enable the electronic nose to deconvolute target vapors from an unknown background, either by physical or chemical separation or other means. With a fully developed molecular model of sensor response to analytes, it may be possible to construct the chemical structure of a compound whose response pattern has not been previously recorded, much as traditional analytical techniques build up the chemical structure of an analyte from its physical or chemical characteristics.7

The immediate application of an electronic nose to the space program is in realtime, continuous air-quality monitoring in human habitats. The ability to monitor recycled breathing air is important for crew health and safety in the living and working quarters of a space habitat such as the International Space Station and in crew vehicles (e.g., the space shuttle or the new crew exploration vehicle now under development by NASA). This ability will also be important in future lunar or martian surface habitats. Today, there is no real-time airquality monitoring in the International Space Station. Air quality is determined anecdotally by crew members' reports and is measured by collecting samples for return to a terrestrial laboratory for analysis using high-sensitivity analytical techniques such as gas chromatography/mass spectrometry (GC/MS) or gas chromatography/ion mobility spectrometry (GC/IMS).

A real-time air-quality monitor would be used as an incident monitor. The crew would be notified of the presence of compounds that are approaching dangerous levels so that remedial action could be taken, or, in a fully automated system, remediation would be part of an automated environmental monitoring and control system.<sup>4,8,9</sup>

The specifications for an incident monitor to be used in spacecraft are that it be capable of identifying and quantifying target com-

pounds at determined levels over a fairly wide range; that it be a low-mass and lowvolume device that requires low power for continuous operation; and that it require little crew time for maintenance, calibration, and air analysis. These qualities are found in electronic noses, and the JPL ENose has been developed to meet these requirements. It can identify and quantify compounds in its target set with a dynamic range from about 0.01 ppm to 10,000 ppm, depending on the compound; it lends itself to miniaturization and low-power operation; and because it measures deviation from a background, it does not require frequent calibration and maintenance.

The JPL ENose is trained to identify and quantify compounds at the 24 h SMAC to provide early warning if a compound is approaching a dangerous concentration. In developing the JPL ENose, the 24 h SMAC was selected as the target concentration because environmental monitoring in crew quarters is envisioned to consist of several devices used at varying intervals.10 A complete picture of spacecraft air will be generated by on-site analysis using a more complex, high-sensitivity analytical device such as GC/MS or GC/IMS with a frequency of once a day to every few days. An incident monitor such as the electronic nose is designed to monitor for changes caused by leaks, spills, or incipient fires between more detailed analyses.<sup>1,3,8,9</sup> In Table I, the analytes selected as target compounds for the JPL ENose are listed, along with their 24 h SMACs.<sup>11</sup> The JPL ENose has been trained to identify and quantify these compounds at concentrations from one-third to three times the 24 h SMAC.

# The JPL ENose

The JPL ENose is a complete, autonomous instrument composed of five basic parts:

- Thirty-two chemical sensors: polymercarbon black composite films on ceramic substrates.
- Electronics designed to operate the device and to acquire and store data.
- A pneumatic system, including a pump and filter system to bring air into the sensing chamber and to provide cleaned air for baselining, and a flow system designed to provide air in turbulent flow to ensure good mixing in the sampled air.
- The data analysis system, consisting of software comprising the training library designed to identify and quantify compounds.
- The computer that controls the device operation, acquires and stores data, processes the data, and provides a readout of results.

The first-generation ENose, built for the 1998 flight experiment, is shown in Figure 1a. It occupies a volume of ~2000 cm³, has

Table I: Target Compounds and Their 24 h Spacecraft Maximum Allowable Concentration (SMAC) as Recorded in the Training Library for the JPL Electronic Nose.

SMAC, 24 h (ppm)
6
270
4
30
10
150
10
35
2000
50
0.3
1
5300
30
0.002
400
40
11
16
100

*Note*: The JPL ENose has been trained to identify and quantify these compounds at concentrations from one-third to three times the 24 h SMAC.

a mass of  $\sim$ 1.5 kg, and uses 1.5 W average power (3 W peak power).<sup>1,3</sup> The secondgeneration ENose (Figure 1b) has the same functions as the first-generation device, but has been miniaturized to occupy ~1000 cm<sup>3</sup> with a mass of  $\sim$ 900 g. The power requirements of the second-generation ENose are similar to those of the first-generation device, even with the switch from the palmtop computer shown in Figure 1a to a PDA-type computer, but this may change if a different computer is eventually chosen. The body and flow system of the second-generation device are made from a single block of hard-anodized aluminum; this design was chosen to eliminate fittings and ensure that there are no leaks in the flow system.

# Polymer–Carbon Black Composite Sensors and the Sensing Array Sensors

The sensors used in the JPL ENose are conductometric, made from polymer–carbon black composite films (a sensor is conductometric when the transduction method is measurement of the dc resistance change in the sensor in response to a target compound). The sensors are made by

solvent-casting polymer-carbon black composite films onto screen-printed Pd-Au electrodes on a ceramic substrate. A photograph of one sensor substrate, consisting of eight sensors, is shown in Figure 2.1 The sensor substrate includes RuO<sub>2</sub> heaters on the back side for temperature control of the sensors during operation. Films are made by dispersing carbon black in a solution containing dissolved insulating polymer, then dispensing the solution onto the substrate. When the solvent has evaporated, the films are electronically conducting because of conductive pathways formed by the carbon black in the polymer matrix. 12 We are currently doing work to determine the optimum concentration of carbon black above the critical amount, or percolation threshold. The carbon black concentration must be sufficient to ensure formation of continuous conduction pathways without increasing the density or thickness of the film too much in order to detect low concentrations of

Responses in the sensors are read as changes in dc resistance. A baseline resistance is established for each sensor, and changes in that resistance indicate a change in the vapor constituents of the environment. The mechanism of response in polymercarbon black composite sensing films is, at its simplest level, based on swelling in the film. Vapors in the environment sorb to the film, causing the film to swell. Swelling disrupts conductive pathways in the film by pushing carbon particles apart, and the electronic resistance in the film increases. 1,2,13 This increased resistance is measured and is used to construct the pattern of response across the sensing array (the "fingerprint" or "signature"), which is analyzed. Other mechanisms, such as ionization of analyte in water trapped in the film or sorption to carbon particles and interrupting conductive pathways, may also contribute to a change in resistance in polymer-carbon black composite sensors. It is elucidation of these mechanisms and the chemical and physical characteristics that govern them that are the focus of the molecular modeling work under way at JPL.5,7

Because the polymer swelling begins immediately after exposure to the vapor, the resistance signals can be read in real time or near real time. With films on the order of 0.1–0.2  $\mu$ m thick, the swelling (and therefore resistance) response times to equilibrium film swelling values range from <0.1 s to >100 s, depending on the vapor and the polymer through which the vapor must diffuse.

At small swellings, the film returns fully to its initial unswollen state after the vapor source is removed, and the film resistance on each array element returns to (or

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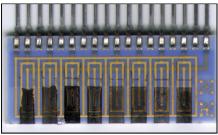
Figure 1. The JPL ENose. (a) First-generation device, used in space shuttle flight STS-95 experiment in 1998 (ruler, 10 cm); (b) second-generation device (ruler, 15 cm).

very near to) its original value. Reversibility, or the tendency of sorbed vapors to leave the surface after the vapor source is removed, has been demonstrated for the polymer composite films used in our arrays for many thousands of vapor exposures, under a variety of ambient conditions in room air at various relative humidities and temperatures, for a diverse set of odors and polymers. 1,14 Such reversibility leaves the sensor surface free to detect the target molecule again, increasing the sensor's lifetime.

### Selecting the Sensing Array

The sensing array used in the JPL ENose is a 32-element array made up of four substrates, each with eight sensors of the type shown in Figure 2. Sensors made from 16 polymers are cast on these substrates. The polymers selected as elements in the JPL ENose chemical-sensing array represent a wide variety of chemical functionalities, because the analyte set to be detected represents a wide variety of compounds.

To obtain distinctive patterns across the array, polymers for the second-generation



2.5 cm

Figure 2. JPL ENose sensor substrate, a 25-mm-wide ceramic base with eight screen-printed Pd-Au electrode sets, used in both devices in Figure 1. Polymer–carbon black composite films are deposited from solution on the electrodes. Heaters have been screen-printed on the back side of the ceramic.

ENose were selected according to a three-step process:<sup>1</sup>

First, polymers were selected to represent five categories of chemical functionality: hydrogen-bond acidic (HBA), hydrogen-bond basic (HBB), dipolar and hydrogen-bond basic (D-HBB), moderately dipolar and weakly H-bond basic or acidic (MD-HB), and weakly dipolar with weak or no hydrogen-bond properties (WD). Responses of these polymers to a subset of target analytes were measured to provide data for further analysis.

Second, a statistical analysis of measured responses of polymers to representative analytes (selected by functional group) was used to determine the best distribution of patterns of response across the analytes. The selection of the sensor set and their corresponding statistical weights were optimized by maximizing distances between gas signatures. Distance between signatures is defined as

$$\Delta S_{mn} = \frac{1}{N} \sum_{i}^{N} |dR_m(i) - dR_n(i)|, \qquad (1)$$

where  $dR_m(i)$  is the (fractional) resistance change of the ith sensor for the mth gas, and the summation is over N sensors used. The gas signature is the unique pattern formed by response of the sensor array to a particular analyte and is generally shown as a histogram of the normalized magnitude of response of each sensor in the array.

Third, a set of polymers was selected from the polymers indicated by statistical analysis as providing a high degree of definition from one analyte to the next. In the final selection, polymer films that showed the lowest-noise response and a high degree of reproducibility of response were chosen.

The polymers used in the first-generation ENose were selected based only on statistical analysis; chemical functionality was not considered. The polymers used in each device and their functionality classifications are shown in Table II.

Polymer selection depends greatly on the suite of target compounds. If the target compounds represent a broad distribution of chemical functionalities, as for the JPL ENose, then a broad distribution of polymers will be necessary. Figure 3 shows that the same set of polymers will distinsignificantly different using the 32-sensor

guish compounds of very similar structures (isomers),6 as well as compounds of very different chemical structures, for instance, propanol and benzene.<sup>3</sup> Figures 3a and 3c show that propanol isomers can be distinguished using 16 polymer-carbon black composite sensors; Figures 3b and 3d show that the benzene and toluene patterns are

Table II: Polymers Used in First- and Second-Generation Electronic Noses and Their Chemical Functionality Classification.

Polymer	Classification	Position in Array
First-Generation Polymers		·
Poly(4-vinylphenol)	HBA	3, 4
Methyl vinyl ether/maleic acid		
50/50 copolymer	HBA	29, 30
Polyamide resin	HBB	7, 8
Poly(N-vinyl pyrrolidone)	HBB	23, 24
Poly(2-hydroxyethyl methacrylate)	D-HBB	11, 12
Vinyl alcohol/vinyl butyral, 20/80	D-HBB	13, 14
Hydroxypropyl methyl cellulose, 10/30	D-HBB	31, 32
Poly(2,4,6-tribromostyrene), 66%	MD-HB	1, 2
Cellulose triacetate	MD-HB	9, 10
Poly(caprolactone)	MD-HB	15, 16
Poly(vinyl chloride-co-vinyl acetate),		
90/10	MD-HB	19, 20
Poly(vinyl acetate)	MD-HB	21, 22
Poly(vinyl stearate)	MD-HB	27, 28
Poly(ethylene oxide)	WD	5, 6
Poly-α-methyl styrene	WD	17, 18
Styrene/isoprene, 14/86 ABA Block		
copolymer	WD	25, 26
Second-Generation Polymers		
Poly(4-vinylphenol-co-methyl methacryla	ate) HBA	1, 9
Poly(ethylene-i-acrylic acid), 80/20	HBA	2, 10
Poly(styrene-i-maleic acid)	HBA	3, 11
Poly(N-vinyl pyrrolidone)	HBB	4, 12
Poly(2-vinyl pyridine)	HBB	5, 13
Poly(4-vinyl pyridine)	HBB	6, 14
Vinyl alcohol/vinyl butyral copolymer	D-HBB	7, 15
Cyanoethyl hydroxyethyl cellulose	D-HBB	8, 16
Ethyl cellulose	D-HBB	17, 25
Poly(vinylbenzyl chloride)	D-HBB	18, 26
Soluble polyimide (matrimide TM 5218)	MD-HB	19, 27
Polyepichlorohydrin	MD-HB	20, 28
Poly(epichlorohydrin-co-ethylene oxide)	MD-HB	21, 29
Ethylene-propylene diene terpolymer	WD	22, 30
Polystyrene	WD	23, 31
Poly(ethylene oxide)	WD	24, 32

<sup>&</sup>lt;sup>a</sup> The position in the array, from 1 to 32, is listed for reference to data in Figure 3. HBA = hydrogen-bond acidic; HBB = hydrogen-bond basic; D-HBB = dipolar and hydrogen-bond basic; MD-HB = moderately dipolar and weakly H-bond basic or acidic; WD = weakly dipolar with weak or no hydrogen-bond properties.

array in the first-generation JPL ENose. The polymers are listed by sensor number in Table II.

By selection of chiral polymers, it has also been shown that it is possible to differentiate enantiomers. 6,15 For example, a poly(R-3-hydroxybutyrate-co-R-3hydroxyvalerate)(77% butyrate)/carbon black composite detector exhibited a differential response to the mirror-image isomers R-2-butanol relative to S-2-butanol. Similar differentiation was observed between enantiomers of α-pinene, epichlorohydrin, and methyl-2-chloropropionate. Each enantiomer of a pair produced a unique relative differential resistance change on sensors made from chiral polymer-carbon black composite film, whereas those same enantiomers produced identical responses on achiral detectors. 15 This type of broadly responsive differentiation to isomers and enantiomers might be useful in probing for optically active signatures of extinct (or even extant) extraterrestrial life as well as in monitoring the quality of spacecraft breathing air.

### Model of Sensor Response

In an effort to understand the chemical and physical characteristics that influence the analyte to sorb to the film, with the further goal of developing the ability to predict the response of any polymer film to any analyte based on its molecular structure, we have initiated an effort to construct a model of sensor response.

Previous authors have shown that studies of linear solvation energy relationships (LSERs) of analytes to polymers can be used to predict the response of polymer films used as receptors in surface acoustic wave sensors. 16,17 Ŝimilarly, such studies should shed light on the response of sensors based on polymer-carbon black composites. However, carbon black particles change the physical characteristics of the film (glasstransition temperature, rigidity, and density), and possibly the chemical characteristics (analytes can sorb or bind to the carbon surface as well as in the film, and sorption sites in the polymer may be occupied by carbon), so solvation energies may not be sufficient for complete description of the sensor-analyte interaction. Neither do LSERs account for analyte molecule diffusion within the film, film thickness, film hydration, or interactions between analyte molecules.5,7

We have developed a model of sensing film-analyte interaction that takes into account the interaction energies of the polymer, carbon black, analyte, and water. Modeling is done using Cerius<sup>2</sup> software. To construct a polymer-carbon black composite model<sup>7</sup> based on the experimental formulations, a polymer box of appropriate

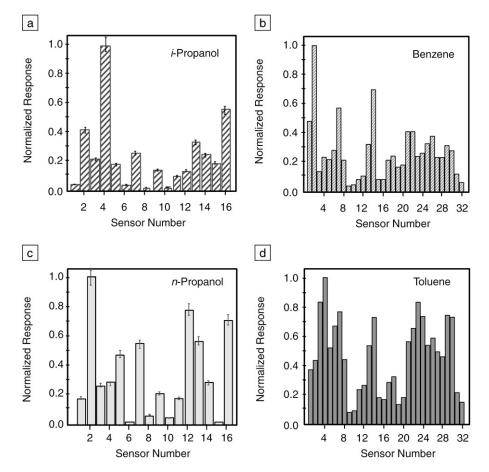


Figure 3. Fingerprints of 60 ppm (a) i-propanol and (c) n-propanol to 16 polymers. Sensor response as  $dR/R_0$  is normalized to the largest response for each array fingerprint. The fingerprints are distinct enough to distinguish the isomers of propanol. Sensor numbers refer to polymers in the odd-numbered positions in the first-generation array (Table II). The response patterns across the array of the polymer—carbon black composite sensors used in the first-generation JPL ENose are significantly different for compounds of similar chemical structure, (b) benzene and (d) toluene. Sensor numbers refer to polymers in those positions in the first-generation array (Table II).

density is created, then carbon black modeled as uncharged naphthalene rings (with no hydrogen atoms) is inserted into the box using a random cavity search in the polymer matrix; naphthalene mimics the hexagonal shape of carbon black particles in the event that shape is important in the sensor—analyte interactions. An equilibrium composite structure is obtained using a combination of molecular mechanics and molecular dynamics simulation techniques.

When the polymer–carbon black composite is equilibrated, interaction energies between one analyte molecule and the composite are calculated using the SORPTION module in the Cerius² software (at 300 K). The contributions to the total interaction energy between an analyte and the composite are its interactions with the polymer chains, carbon black, and other compounds (such as water) that may be present. The

simulation program generates random points in the composite model and attempts to insert the analyte molecules. Insertion attempts that involve the overlapping of the analyte molecule with the composite structure are discarded. For each composite–analyte interaction, 2–3 million simulations (analyte insertions) were run, and the average energy of composite–analyte interactions was calculated at the end of the simulation.

Figure 4 shows the predicted interaction energies of a selection of target analytes with modeled composites.<sup>7</sup> Work is in progress to validate the model through experimental work and to describe the response mechanisms by correlating sensor responses to molecular descriptors based on quantitative structure–activity relationship (QSAR) techniques using so-called genetic function approximations. As used in this

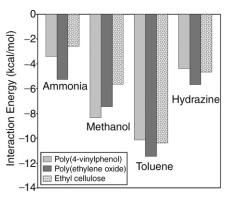


Figure 4. Predicted target analyte interaction energies with polymer–carbon black composites.

work, QSAR correlates the response (activity) of the sensor to several physical and chemical properties stemming from the molecular structure of both the analyte and the sensor (molecular descriptors). Examples of molecular descriptors used in this work are the calculated energy of interaction between analyte and polymer and between analyte and carbon black, the glass-transition temperature of the polymer, and the molecular volume of the analyte.

# Effect of Environmental Conditions on Sensors

As with most chemical sensors, local conditions can have a significant effect on the response of polymer-carbon black composite films used as sensors. Whereas changes in humidity, temperature, and pressure tend to occur on a relatively slow time scale and so can be ignored if the device is used to monitor changes over the previous few minutes, we have designed the JPL ENose to be able to look back over long periods (several hours) to determine whether there is evidence of a slow buildup of target compounds. On this time scale, changes in environmental conditions can obscure small changes in sensor response, because the sensors are very sensitive to changes in relative humidity of 1% or less and temperature changes of ~0.2°C.1,3 To diminish the effect of local temperature variations, the sensor substrates used in the JPL ENose are heated to a target temperature that is 1-4°C above the ambient temperature; target temperatures of 24°C, 28°C, 32°C, or 36°C may be used, depending on the local ambient temperature. The response of the sensor to each target compound as a function of temperature has been characterized. Humidity is measured independently of the polymer-carbon black composite sensors and taken into account in data analysis through empirically determined algorithms

that describe the effect of humidity on each analyte in each polymer.

To distinguish between slow changes in sensor resistance caused by a slow leak of a target compound and those caused by baseline drift, a protocol for baselining was included in the ENose. This protocol involves switching the air intake through a carbon filter that removes volatile compounds to provide cleaned air to the sensors at set intervals, then analyzing the virtual peak formed when the flow switches back to ambient air. The peak is "virtual" because it is caused by an artificial event created by the experimenters. If there is no change in the magnitude or fingerprint of the virtual peak over time, the air is considered to be unchanged and safe. If there is a change in the magnitude or the fingerprint of the virtual peak, the data analysis program attempts to identify the compound responsible for the change.1,3

# **Data Analysis**

The primary constraint in data analysis software development was the requirement that gas events of single or mixed gases from the target compounds be identified correctly and quantified accurately. (Quantification accuracy was required to be only ±50% of known concentration because toxicological quantities are not better known than 50%.) In addition, three conditions were found in the data expected for air-quality monitoring: a large number of target compounds, some of which are of a very similar chemical structure (e.g., benzene and toluene); low target concentrations with nonlinear responses for some sensors at low concentrations; and single gases and mixtures.

To address these considerations, we have designed an automated routine to identify and quantify a gas event using an algorithm based on Levenberg–Marquart nonlinear least-squares fitting (LM-NLS).<sup>1,3,18</sup> Several other pattern recognition approaches were considered before LM-NLS was selected.<sup>1,3</sup>

In the first-generation ENose, the success rate for identification and quantification was 85%. This success rate takes account of false positives and negatives as well as incorrect identification or quantification.<sup>1,3</sup> Further work is improving this rate.

### **Conclusions**

The device developed as the secondgeneration ENose at JPL has been tested for its ability to respond to the compounds in Table I and is ready to operate as an autonomous device. Work continues in developing a model of sensor—analyte interaction to optimize the selection of materials for a sensing array as well as for possible future use to identify array responses from untrained-for compounds, that is, compounds causing array response patterns not found in the training library.

Future application of the JPL ENose in space habitats or planetary exploration will depend on NASA's needs, but eventual deployment of the ENose as one part of a fully automated environmental monitoring and control system is envisioned. In this scenario, activities such as remediation of spills and leaks would be actuated through a logic system and performed robotically.

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